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भारतीय मानक

लिन्डेन डस्ट पाउडर — विशिष्टि

Indian Standard

LINDANE DUSTING POWDER — SPECIFICATION

ICS 19.020;65.100.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 2000

Price Group 3

Pesticides Sectional Committee, FAD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

This formulation is based on Lindane (CAS No. 58-89-9) a broad spectrum, pesticide. (Gamma Isomer of HCH having minimum purity of 99.5 percent, almost free from Alpha, Beta, Delta, etc, isomers is called Lindane).

Lindane dusting powders are widely used for control of large number of pests in agriculture, public health at low rate of application.

Lindane dusting powders are generally manufactured to contain 0.65 or 1.3 percent Lindane content (m/m) as active ingredients.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act,* 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

LINDANE DUSTING POWDER — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirement and the method of sampling and tests for Lindane dusting powders.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves : Part 1 Wire cloth test sieves (*third revision*)
882 : 1984	Specifications for Lindane
1070 : 1992	Reagent grade water (*third revision*)
6940 : 1982	Method of test for pesticides : Part 1 Solid pesticides (*second revision*)
8190 (Part 1) : 1988	Requirement for packing of pesticides : Part 1 Solid pesticides
10627 : 1983	Method for sampling of pesticidal formulations

3 REQUIREMENTS

3.1 Description

The material shall consist of a homogeneous mixture of Lindane in a filler and shall be in the form of a fine free flowing powder, free from extraneous impurities or added modifying agents. The powder when dusted from a hand dusting apparatus shall issue freely without clogging and bridging.

3.1.1 Lindane used in the manufacturing of dusting powder shall conform to IS 882 with up to date amendments.

3.2 The material shall also comply with the requirements given in Table 1.

3.2.1 *Lindane Content*

When determined by the method prescribed in Annex A, the observed Lindane content, percent by mass of any of the samples shall not differ from the declared nominal value by more than the tolerance limits indicated below:

Nominal Value, Percent	*Tolerance Limit*	
Up to 9	+ 10 – 5	percent of the nominal value
10 and below 50	± 5	
50 and above	+ 5 – 3	

3.2.1.1 The actual value of Lindane content shall be calculated to fourth decimal place and then rounded off to the second decimal place before applying the tolerances as stipulated above in **3.4.1**.

3.2.1.2 The average content of all samples taken shall not be lower than nominal content.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in mild steel or tin plate or fibre board containers or double hessian Jute bag (*see* IS 8115) or DW tarpaulin laminated jute bags (*see* IS 8117) or HDPE woven sacks/bags (*see* IS 8069) shall be provided with an internal loose polythene liner of thickness not less than 0.062 mm and in the form of tubular sheath bottom sealed.

The general requirement of packaging shall conform to 2 of IS 8190 (Part 1) and amendment thereof, if any.

4.2 Marking

The container shall bear legibly and indelibly the following information in addition to the information as required under the *Insecticide Act* and Rules:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture and date of expiry;

d) Batch number;

e) Net mass of contents;

f) Nominal Lindane content, percent (m/m); and

g) The cautionary notice as worded in *Insecticides Act* and Rules.

4.2.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4.2.2 When bags (*see* **4.1**) are used for packing this material the pictorial marking for 'USE NO HOOKS AND DO NOT PUNCTURE' as contained in IS 1260 (Part 2), shall be stencilled on each bag.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standards Method for sampling of pesticides and their formulations' IS 10627.

6 TESTS

6.1 Test shall be carried out by the methods referred to in col 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis

Table 1 Requirements for Lindane Dusting Powders

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of This Standard	Cl No. of IS 6940
(1)	(2)	(3)	(4)	(5)
i)	Lindane content, percent by mass	Nominal value as declared on the container (*see* 3.4.1)	A	–
ii)	Material passing through 75 micron IS sieve) * percent by mass, *Min*	90	–	12.1
iii)	Bulk density after compacting	Not to exceed the value obtained before compacting by more than 60 percent	–	12.2
iv)	Acidity (as HCl), percent by mass, *Max* *OR* Alkalinity (as NaOH) percent by mass, *Max*	0.25 0.04	B	–
v)	Related isomers	To pass the test	C	–

* See IS 460 : 1985 Specification for test sieves (*revised*). BS test Sieve 200, ASTM Test Sieve 200, Tyler Test Sieve 200 have their aperture within the limits specified for the above IS Test Sieve, and may, therefore, be used as 75 micron IS Test Sieve.

ANNEX A

[*Table* 1, *Sl No.* (i); and *Clause* 3.2.1]

METHOD FOR DETERMINATION OF LINDANE CONTENT IN LINDANE DUSTING POWDER

A-0 GENERAL

A-0.1 Lindane content is determined by gas-liquid chromatography by using an internal standard and a flame ionisation detector.

A-1 GAS CHROMATOGRAPHIC METHOD

A-1.1 Gas liquid chromatograph fitted with a flame ionisation detector (FID) is used for this determination. Using solution containing known amounts of the standard Lindane and internal standard the response factor *R* for Lindane content and internal standard is arrived at. A solution containing a known mass of sample and internal standard is injected subsequently into the GLC unit. The percentage of Lindane content is then computed by standard relationship.

A-1.2 Apparatus

A-1.2.1 *Gas Liquid Chromatograph*

The instrument should be one that is designed for use with glass columns and that is equipped with an on-column injection system and a high-sensitivity flame ionization detector. The suggested parameters are given in the operating conditions (**A-1.5.1**) which can be changed depending upon the available equipment and allied facilities provided standardisation is done.

Chromatographic Column— The column should be a borosilicate glass tube 183 cm length and 4 mm i.d., packed with 5 percent OV-210 on chromosorb W-IIP (100-120 mesh) or suitable equivalent, bent to fit the chromatograph.

A-1.2.2 *Microsyringe*

10 micro litre capacity

A-1.3 Reagents

A-1.3.1 *Acetone*

Analytical reagent grade

A-1.3.2 *Lindane Standard of Known Purity*

A-1.3.3 *Internal Standard*

Alpha naphthol

A-1.4 Preparation of Standard and Sample Solutions

A-1.4.1 *Preparation of Internal Standard Solution*

Weigh accurately about 150 mg of internal standard into a 100 ml volumetric flask and make up the volume using acetone. This will contain 1.5 mg/ml of the internal standard.

A-1.4.2 *Preparation of Standard Lindane Solution*

Weigh accurately about 100 mg standard Lindane into a 100 ml, flask with glass stopper and add by pipette 25 ml of internal standard solution (**A-1.4.1**) and 25 ml acetone. Shake for a minute to dissolve Lindane.

A-1.4.3 *Preparation of Sample Solution*

A-1.4.3.1 Weigh accurately a quantity (Sample quantity for 0.65 percent DP-approx. 6 gm, sample quantity for 1.3 percent DP-approx 3 gm) of sample containing about 40 mg of Lindane into a 50 ml flask with glass stopper. Add by pipette 10 ml of internal standard solution and 10 ml acetone.

Stopper the flask tightly and gently swirl the contents for one minute using a rotational motion of the wrist. Allow each flask to stand for at least five minutes and then shake for one minute. Centrifuge the above solution in a stoppered tube and use supernatant layer as sample solution for injection in gas chromatographic column.

A-1.5 Estimation

A-1.5.1 *Operating Conditions*

A-1.5.1.1 *Operating conditions for gas liquid chromatography*

The temperatures, gas flow rates and retention times given below are typical values and may have to be adjusted to obtain optimum results from a given apparatus.

Gas flow

Carrier gas (nitrogen)	25 ml/min
Fuel gas (hydrogen)	30 ml/min
Air	300 ml/min

Temperature

Column oven	160°C
Injection port	220°C
Detector	240°C
Chart speed	0.5 cm/min
Injection size	2 micro litre

Typical Retention Times:

Alpha-naphthol (I.S.) peak	2.7 min
Lindane peak	6.6 min

A-1.5.1.2 Inject 2.0 (l portions of standard Lindane solution until response factor (peak area) for Lindane to internal standard agrees to within two percent.

A-1.5.3 Inject duplicate aliquots of the sample solution (**A-1.4.3.1**) using same column as that of standard. Measure the response factors and compute Lindane content.

A-1.6 Calculations

$$\text{Response factor } (R) = \frac{\text{Area of lindane peak}}{\text{Area of internal standard peak}}$$

$$\text{Lindane content, percent by mass} = \frac{R \times W^1 \times P}{R^1 \times W \times 2.5}$$

where

R = response factor for sample solution,

R^1 = response factor for standard solution,

W = weight of sample,

W^1 = weight of Lindane standard, and

P = percent purity of Lindane standard.

NOTE — A typical chromatogram for the internal standard and standard Lindane solution is enclosed for reference.

ANNEX B

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ACIDITY OR ALKALINITY

B-1 QUALITATIVE TEST

B-1.1 Procedure

Take about 0.5 g of the material in a test tube mix with about 1 ml of neutral acetone and warm gently to effect solution of the Lindane present in the material. Cool and add 1 ml water to it and then test the solution for acidity or alkalinity with a litmus paper. Determine the acidity or the alkalinity as the case may be, by the methods prescribed under **B-2** and **B-3**, respectively.

B-2 DETERMINATION OF ACIDITY

B-2.1 Reagents

B-2.1.1 *Acetone*

B-2.1.2 *Methyl Red Indicator Solution*

Aqueous, 1 percent (*m/v*)

B-2.1.3 *Standard Sodium Hydroxide Solution*

0.05 N.

B-2.1.4 *Standard Hydrochloric Acid*

0.05 N.

B-2.2 Procedure

B-2.2.1 Weigh accurately about 10 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to effect solution of the Lindane present in the material. Cool, add 75 ml of water, filter and then titrate immediately with the standard Sodium Hydroxide solution using methyl red as the indicator.

B-2.2.2 Carry out a blank determination using 25 ml acetone diluted with 75 ml of water.

B-2.3 Calculation

$$\text{Acidity (as HCl), percent by mass} = \frac{3.646\,(V-v)\,N}{M}$$

where

V = volume, in ml of the standard sodium hydroxide solution required for the test with the material;

v = volume, in ml of the standard sodium hydroxide solution required for the blank determination;

N = normality of the standard sodium hydroxide solution; and

M = mass in g, of the material taken for the test.

B-2.3.1 In case the blank shows alkaline reaction, neutralize with the standard Hydrochloric Acid and calculate as follows:

$$\text{Acidity as HCl, percent by mass} = \frac{3.646\,(VN_1 + vN_2)}{M}$$

where

V = volume, in ml of the standard hydroxide solution, required for the test with the material;

V = volume in ml of the standard hydrochloric acid required for the blank determination;

N_1 = normality of the standard sodium hydroxide solution;

N_2 = normality of the standard hydrochloric acid; and

M = mass in g, of the material taken for test.

B-3 DETERMINATION OF ALKALINITY

B-3.1 Reagents

B-3.1.1 *Acetone*

B-3.1.2 *Methyl Red Indicator Solution*

Aqueous, 1 percent (*m/v*)

B-3.1.3 *Standard Hydrochloric Acid*

0.02 N.

B-3.1.4 *Standard Sodium Hydroxide Solution*

0.02 N.

B-3.2 Procedure

B-3.2.1 Weigh accurately about 15 g of the material into a dry conical flask, add 40 ml of acetone and mix. Warm the flask gently to effect the solution of the Lindane present in the material. Cool, add 75 ml of water, filter and then titrate immediately with the standard hydochloric acid using methyl red as the indicator.

B-3.2.2 Carry out a blank determination using 40 ml of acetone diluted with 75 ml of water.

B-3.3 Calculation

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{4.0\,(V-v)N}{M}$$

where

V = volume, in ml, of the standard hydrochloric acid required for the test with the material;

v = volume, in ml, of the standard hydrochloric acid required for the blank determination;

N = normality of the standard hydrochloric acid; and

M = mass in g, of the material taken for the test.

B-3.3.1 In case the blank shows acid reaction, neutralise with the standard sodium hydroxide solution and calculate as follows:

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{4.0\,(VN_1 + vN_2)}{M}$$

where

V = volume, in ml, of the standard hydrochloric acid required for the test with the material;

N_1 = normality of the standard hydrochloric acid;

v = volume in ml, of the standard sodium hydroxide solution required for the blank determination;

N_2 = normality of the standard sodium hydroxide solution; and

M = mass in g, of the material taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF RELATED ISOMERS IN LINDANE DUSTING POWDER

C-0 GENERAL

C-0.1 For the checking of presence of other isomers than Lindane, gas chromatogram obtained in Annex A is to be examined.

C-1 GLC METHOD

C-1.1 GLC chromatogram obtained during determination of Lindane (Annex A) should normally show peak corresponding to internal standard and lindane only. Product does not conform to the specification if there are any secondary peaks corresponding to alpha, beta or delta isomers and area of any such peak is more than one percent of the area of Lindane peak.

Typical Retention Time

Alpha-naphthol(I.S.) peak	2.7 min
Alpha-isomer peak	5.5 min
Lindane peak	6.6 min
Beta-isomer peak	8.1 min
Delta-isomer peak	9.0 min

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices : Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110 002
323 76 17
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Eastern : 1/14 C. I.T. Scheme VII M, V. I. P. Road, Kankurgachi
CALCUTTA 700 054
337 84 99, 337 85 61
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Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022
60 38 43
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TO
IS 14834 : 2000 LINDANE DUSTING POWDER — SPECIFICATION

(*Page* 3, *clause* **A-1.5.1.2**, *line* 1) — Substitute '2.0 µl' *for* '2.0(l'.

(*Page* 3, *clause* **A-1.6**, *Note*) — Insert the following at the end of the Note:

'Run the chromatogram for about double the time (approx. 13 min) than the retention time of Lindane (approx 6.6 min) to check the presence of other isomers.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India